Refractive Index of Glass by GRIM

1 Scope

This document provides the procedures for determining the refractive index (RI) of microgram sized glass fragments at up to three wavelengths using the Foster and Freeman, Ltd. Glass Refractive Index Measuring System (GRIM 3) by Geologist/Forensic Examiners within the Trace Evidence Unit (TEU). RI is the ratio of the velocity of speed of light in one media compared to the speed of light in a vacuum. It varies according to the wavelength of the light and the temperature of the medium, but its value in a particular glass at a set wavelength and temperature is a function of the composition of the glass and its thermal history. RI provides excellent discrimination among glasses, and is the most commonly measured property in forensic glass comparisons. Refer to *Glass Refractive Index Determination* by the Scientific Working Group for Materials Analysis (SWGMAT) and ASTM E1967-11a, *Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope* for further information.

Using the GRIM 3 method, a glass fragment is immersed in an appropriate reference liquid and observed at the wavelength of interest while the temperature is electronically varied until the match point temperature is reached. The match point temperature is automatically recorded. The match point temperature can be determined at multiple wavelengths, typically n_D, n_C, and n_F.

2 Equipment/Materials/Reagents

- 1 or 1½ gauge coverslips
- Alcohol (methanol, ethanol, or isopropyl alcohol, any grade)
- Detergent such as sodium hexametaphosphate (or equivalent)
- Forceps
- Glass microscope slides, 76mm x 19mm
- Glass RI reference materials
    - National Bureau of Standards (NBS) melt 9012
    - Bundeskriminalamt (BKA) K5 (Schott Optical Glass)
    - Additional standards at the discretion of the Examiner
- GRIM 3
- Hammer mill
- Hot stage capable of maintaining a temperature to ± 0.2°C or better
- Interference filters, wavelengths approximately n_D, n_C, n_F
- Kimwipes, Techwipes, or equivalent lint-free paper tissue
- Locke Scientific standard reference glasses (Locke B1 through B4, Locke B6 through B12, Locke A1 through A5, Locke C1 and Locke C2, or equivalent)
- Locke Scientific Silicon oil A, B, or C or equivalent
- Nitric acid (HNO_3, any grade)
- Personal Protective Equipment, as needed
- Phase Contrast Microscope with minimum 10x magnification
- Probes (e.g., metal, wooden)
- Ultrasonic bath

- Water

3 Standards and Controls

3.1 Standard glasses of known RI, such as NBS melt 9012 and BKA K5 Schott Optical Glass, are mounted on a glass microscope slide in the appropriate oil. An appropriate oil is similar in RI to the standard glass at the temperature and wavelength of interest. The mounted glass standards are then analyzed by GRIM 3 according to section 2 of the GRIM 3 Glass Refractive Index Measurement System Instruction Manual and User Guide.

3.2 Standards are stored at room temperature and pressure, and maintained in separate containers to prevent contamination. Properly maintained standards have an indefinite lifetime.

3.3 Accuracy of the GRIM 3 system is assured by proper alignment of the instrument. The alignment of the instruments is checked prior to each use. See the Trace Evidence General Microscopy Techniques for instructions on microscope alignment.

3.4 The GRIM 3 will be calibrated: yearly when the instrument is in use; prior to use if not calibrated within the previous 12 months; or if the measurement of the standards exceeds accepted tolerances and cleaning and re-alignment fail to correct this situation.

3.5 Locke Silicon oil (A, B, or C) will be calibrated for the temperature range of interest. The oil chosen is based upon the expected RI of the glass to be analyzed. A minimum of three standard reference glasses will be used when calibrating the Locke A and B oils. Only two standard reference glasses are available for the RI range of the Locke C oil, and both must be used when calibrating the Locke C oil. Additional oils (e.g. Dow Corning 710 or 550 oil) may be calibrated for use at the discretion of the examiner.

3.5 Calibrate the GRIM 3 according to the guidelines presented in the GRIM 3 Glass Refractive Index Measuring System User Manual, Sections 2 and 9.

3.5.1 A calibration curve is constructed for each silicon oil of interest at the n_D, n_C, and n_F wavelengths.

3.5.2 Collect six measurements for each of the standard reference glasses at the temperatures of interest. Do not use the Locke Scientific Standard Glass B5.

4 Sampling or Sample Selection

4.1 For known source items, several samples should be selected to represent the range of RI measurements of the glass (Sandercock, 2000; Garvin and Koons, 2011).

4.1.1 When sufficient glass is available, select at least seven fragments from the known sample to mount on individual glass microscope slides. If there are less than seven fragments in a known sample, make at least one glass microscope slide mount per fragment.

4.1.2 Measure at least 21 different suitable edges, with an approximately equal number of

measurements made per slide produced. To avoid bias, select the first suitable edges found upon scanning the slide.

4.1.2.1 Suitable edges are bright, thin, clean, smooth and sharp (see the GRIM 3 Glass Refractive Index Measuring System User Manual, Sections 2 and 7). The edge traces provide an indicator of acceptability. Fragments with poor edge traces will be assessed as to their suitability.

4.2 When measuring RI of questioned glass, measure all suitable fragments that have not been previously disassociated by other tests, or a minimum of ten fragments in cases where there are more than ten suitable fragments that have not been previously disassociated by other tests. If all of these fragments are disassociated by RI measurement, additional glass, if present and suitable, will be measured. The total number of glass fragments measured is at the discretion of the examiner.

4.2.1 Suitable glass fragments are those of sufficient size to crush for measurement of fresh edges. Additional factors such as the presence of coatings or contaminants may limit suitability. The determination of suitability of each glass fragment is left to the discretion of the Geologist/Forensic Examiner.

4.2.2 If more than ten glass fragments are recovered from a questioned item, but less than ten glass fragments are suitable for analysis, the reason(s) why the fragments are unsuitable will be recorded in the case notes. If less than ten glass fragments are recovered and any of them are unsuitable for analysis, the reason(s) why the fragments are unsuitable will be recorded in the case notes.

4.2.3 Measure all suitable edges, or a minimum of ten edges in cases where there are more than ten suitable edges.

5 Procedures

5.1 Turn on the computer, monitor, and GRIM 3 power and place the interference filter of the wavelength of interest over the field diaphragm. Allow electronics to warm up for approximately one hour.

5.2 Check that the microscope optics and hot stage are clean and free of oil. Clean if necessary. See Trace Evidence General Microscopy Techniques.

5.3 Arrange the microscope for optimum illumination and phase contrast. To ensure maximum contrast, make sure the annular illumination ring from the condenser is properly aligned with the phase contrast shift plate located within the objective by viewing the superimposition at the back focal plane of the objective. This can be accomplished in a number of ways, the most convenient of which are the use of a Bertrand lens or a phase centering telescope. See Trace Evidence General Microscopy Techniques.

5.4 Clean a glass RI slide with water or alcohol and a clean, dry, lint-free wipe.

5.5 Put a drop of the appropriate Locke Scientific silicon oil A, B, or C on the cleaned

slide. For the majority of soda-lime silicate glasses, Locke Scientific B is the appropriate oil. Locke Scientific C is the appropriate oil for most borosilicate glasses. Locke Scientific A is appropriate for very high refractive index glasses.

5.6 Select a clean particle previously determined to be glass (see Forensic Glass Examinations) and place it in the oil on the slide. If needed, fragments may be cleaned prior to analysis.

5.6.1 Clean fragments by soaking them in alcohol, a detergent solution followed by a water rinse, or nitric acid followed by a water rinse. Fragments may also be cleaned in an ultrasonic bath in alcohol, or in a detergent solution followed by a water rinse. Following cleaning, dry thoroughly.

5.7 Crush the fragment(s) in situ with a clean metal probe or equivalent. Glass fragments can also be crushed in a clean hammer mill or equivalent and transferred to the glass microscope slide. Fragments too small to be crushed by these methods are inappropriate for analysis.

5.8 Heat slide for approximately 15 minutes at 100°C to drive off volatile impurities in the oil.

5.9 Cover sample with a clean coverslip. In necessary, coverslips may be cleaned using alcohol and a lint-free wipe.

5.10 Create a case according to the GRIM 3 Glass Refractive Index Measuring System User Manual, using the unique FBI case and item identifiers.

5.11 Measure the refractive index of the glass standards (NBS 9012 and BKA K5), followed by the glass specimens from the case. If measurement of the case specimens exceeds one day, the glass standards must be measured each day prior to obtaining measurements for case specimens.

5.11.1 Insert a prepared mount of a glass sample into the hot stage mounted on the phase contrast microscope stage and allow the sample and hot stage to equilibrate.

5.11.2 Check the alignment of the microscope and adjust if necessary. See Trace Evidence General Microscopy Techniques.

5.11.3 Locate the first suitable fragment found upon scanning the slide, and position an edge box on the edge. The GRIM 3 can measure up to four edges simultaneously. A focus indicator appears as a three digit number in the bottom right hand corner of the video window. The focus of the fragment must be adjusted so that the focus indicator is at its maximum prior to a measurement. When analyzing multiple edges, if the focus indicator cannot be maximized for all edges simultaneously, then only edges that can be maximized may be measured in a single run.

5.11.4 Adjust the temperature of the hot stage so that the temperature is slightly above the match point of the glass. Two degrees above the match point is recommended.

5.11.5 Analyze the glass fragments according to section 2 of the GRIM 3 Glass Refractive

Index Measuring System User Manual.

5.11.5.1 Analyze at least five fragments each of two known glass standards (NBS 9012 and BKA K5) at the wavelength of interest prior to each use of the GRIM 3.

5.11.5.1.1 The average value of each standard must be within ±0.00005 of the accepted value. Individual measurements cannot exceed ±0.0001 of the accepted value.

5.11.5.1.2 If the average value of either of the standard glass falls outside of the accepted tolerance (±0.00005), or the individual measurements exceed ±0.0001 of the accepted value, the GRIM 3 will be cleaned and re-aligned and the standard glasses will be reanalyzed.

5.11.5.1.3 If the average measurement of either of the glass standards falls outside of the accepted tolerance (±0.00005), or the individual measurements exceed ±0.0001 of the accepted value, and cleaning and re-alignment fail to correct this, the instrument will be recalibrated.

5.11.5.1.4 If the average measurement of either of the glass standards falls outside of the accepted tolerance after recalibration, the data collected cannot be used for comparison against any other data collected during a different analysis session.

5.11.5.2 For known glass specimens, measure at least 21 different suitable edges, with an approximately equal number of measurements made per slide if more than one slide was produced.

5.11.5.3 For questioned glass specimens, measure all suitable edges, or a minimum of ten suitable edges in cases where there are more than ten suitable edges. If there are less than three suitable edges, the specimen is inappropriate for analysis using the GRIM 3.

5.11.6 Repeat the above procedure if desired at additional wavelengths by using the appropriate filters.

5.12 At the completion of the analysis, include the results in the case notes.

5.13 If the analysis is a comparison between known source(s) and questioned glass fragments, the resultant range of refractive index values from known broken glass source(s) versus the average of multiple measurements of the questioned glass fragments are used as the comparison criteria. When the average of multiple refractive index measurements of the questioned items falls within the range (≥ minimum and ≤ maximum) of refractive index values of the items from known source(s), the glasses are said to be indistinguishable.

5.14 If the analysis is a comparison between two or more questioned glass fragments, range overlap of multiple measurements of each questioned glass fragments is used as the comparison criteria. When the ranges of one questioned glass fragment (≥ minimum and ≤ maximum) and another questioned glass fragment (≥ minimum and ≤ maximum) overlap, the glasses are said to be indistinguishable.

6 Calculations

An RI average is calculated by summing the RI values for a single item at one wavelength and dividing by the total number of RI measurements for that item at that wavelength.

7 Measurement Uncertainty

The calculated precision of the GRIM 3 is based on the reported precision of the hot stage (0.2ºC) and is between 0.00004 and 0.00007 at all wavelengths.

8 Limitations

8.1 The precision of the method, as determined by multiple measurements of homogenous standard glass samples, is better than the variation of RI in most sample glasses. Typical values of uncertainty in measured indices are in the fifth decimal place.

8.2 Fragments that do not provide a good edge for index determination can result in degraded precision. The quality of the edge trace can be an indicator of the acceptability of the measurement (see GRIM 3 Glass Refractive Index Measuring System User Manual, page 38). RI values from poor quality fragments should not be reported.

9 Safety

Eye protection and gloves will be worn when breaking glass items.

10 References

- ASTM International. ASTM E1967-11a. Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil mmersion Method and a Phase Contrast Microscope. West Conshohocken, Pa.: ASTM International, 2011.
- Garvin, EJ and Koons, RD. Evaluation of Match Criteria Used for the Comparison of Refractive Index of Glass Fragments. Journal of Forensic Sciences March 2011;56(2):491-500.
- Foster and Freeman, Ltd. GRIM 3 Glass Refractive Index Measurement System Instruction Manual and User Guide. July, 2009.
- Laboratory Division Safety Manual (current revision).
- Locke Scientific. Reference Glasses and Silicone Oils for Refractive Index Determination.

Sandercock, PML. Sample Size Considerations for Control Glass in Casework. Canadian Society Forensic Science Journal 2000;33:169-178.

- Scientific Working Group for Materials Analysis (SWGMAT). Glass Refractive Index Determination. Forensic Science Communications January, 2005;7(1), http://www.fbi.gov/hq/lab/fsc/backissue/jan2005/standards/2005standards7.htm, accessed 1/25/2018.

- Trace Evidence General Microscopy Techniques, Trace Evidence Procedures Manual (current version)

Rev. #	Issue Date	History
1	11/02/15	Section 2 Updated list. Section 3 Reworded for clarity. Section 4.1 Added statement about cleaning and realigning prior to redoing calibration. Section 4.2 Reworded for clarity, added possibility of using additional oils. Section 5.1.2 Clarified number of analyses. Section 5.2 Reworded for clarity. Section 5.2.1 Reworded for clarity. Section 5.2.3 Added to clarity number of required measurements. Section 6 Revised and renumbered for clarity and transparency. Section 7 Added calculation of average RI. Updated references. Updated for FA, changing "specimen" to "item" when appropriate.
2	02/07/18	Updated throughout removing references to TEU where appropriate. Added Geologist/Forensic Examiner to the Scope in Section 1 and throughout document where appropriate. In Section 1, added clarification regarding RI ratio. Removed Calibration Section 4 and renumbered. Text previously appearing in Calibration section added to Section 3. Added 'Sample Selection' to new Section 4. 'Recorded' substituted for 'documented' throughout document. References updated in Section 10 and throughout document.

Redacted - Signatures on File

Approval

Trace Evidence Unit Chief — Date: 02/06/2018

Mineralogy Technical Leader — Date: 02/06/2018

QA Approval

Quality Manager — Date: 02/06/2018